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CONSIDERATIONS IN THE MEASUREMENT OF CRYOPUMPING CAPTURE COEFFICIENTS

J. D. Haygood and J. P. Dawson

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CONSIDERATION IN THE MEASUREMENT
OF CRYOPUMPING CAPTURE COEFFICIENTS

J. D. Haygood and J. P. Dawson
ARO, Inc.

FOREWORD

The research reported herein was sponsored by Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee under Program Element 61445014/8951.

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ABSTRACT

A survey is presented of the more important practical and theoretical relationships involved in the study of cryopumping. Starting with a rigorous definition of the capture coefficient, the effects of temperature on the measured values are derived. Several practical points in the design of a chamber for cryopumping measurements are given, including cryosurface and gage placement, calibration, effects of outgassing and impurities, and temperature measurements.

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NOMENCLATURE

A	Cryosurface area, cm^2
C	Capture coefficient
C_g	Condensation coefficient
C_s	Evaporation coefficient
E	Minimum energy, ergs/mole
f	Fraction present
\dot{G}	Rate of mass transfer, gm/sec
k	Boltzmann constant, ergs/deg molecule
LN_2	Liquid nitrogen
M	Molecular weight, gm/mole
m	Weight of a molecule, gm/molecule
N	Number of moles
\dot{N}	Flow rate, moles/sec
\dot{n}	Flow rate, molecules/sec
n/n_0	Fraction of molecules with energy greater than E
P	Pressure, mm Hg
\dot{P}	Change in pressure per unit time, mm/sec
P_c	Steady-state chamber pressure, mm Hg
P_F	Pressure at point F, mm Hg (see Section 6.1 of text)
P_g	Steady-state pressure without flow, mm Hg
P_H	Pressure at point H, mm Hg (see Section 6.1 of text)
P_{He}	Partial pressure of helium, mm Hg
P_i	Partial pressure of the "i"th component, mm Hg
P_m	Measured pressure, mm Hg
P_{N_2}	Partial pressure of nitrogen, mm Hg
P_o	Pressure behind the standard leak, mm Hg
P_s	Pressure at cryosurface conditions, mm Hg
R	Universal gas constant, ergs/mole °K

r_F	Ion gage reading at point F (see Section 6.1 of text)
r_H	Ion gage reading at point H (see Section 6.1 of text)
S	Pumping speed, cc/cm ² -sec
S_{max}	Theoretical maximum specific pumping speed, cc/cm ² -sec
T	Temperature, °K
T_a	Temperature of the analyzer, °K
T_c	Chamber temperature, °K
T_g	Gas temperature, °K
T_o	Room temperature, °K
T_s	Cryosurface temperature, °K
V	Volume, liters
\dot{V}	Pumping speed, cc/sec = AS
V_c	Chamber volume, liters
\dot{V}_{max}	Theoretical maximum pumping speed of cryosurface = $A S_{max}$, cc/sec
\dot{V}_o	Volume flow through standard leak at T_o , P_o , cc/sec
α	Instrument sensitivity factor
α_{He}	Instrument sensitivity factor for helium
α_{N_2}	Instrument sensitivity factor for nitrogen
η	Rate of incidence of molecules on the surface, molecules/sec-cm ²

SUBSCRIPTS

1	}	Pertaining to specific point
2		
A		
B		

SECTION I INTRODUCTION

In the past few years, great advances have been made in vacuum technology. As more experience has been gained, vacuum techniques have become more refined, and understanding of the conditions existing in high vacuum chambers has improved. With improved understanding and techniques, more attention may now be directed toward effects which have been previously hidden in experimental uncertainty.

One of these effects is the variation of cryopumping speed with gas and cryosurface temperatures (Refs. 1, 2, and 3). The pumping speed is of interest in predicting pressures in vacuum systems as a function of the gas load. The capture coefficient, which is derived from the pumping speed, is related to the thermal accommodation coefficient and, indirectly, to the momentum accommodation. Therefore, interest in the capture coefficient extends beyond vacuum technology into low density aerodynamics and surface physics.

It is the purpose of this report to define more rigorously some of the relationships which exist in cryopumping and to point out limitations in the use of these relationships. These relationships may be grouped under the general headings of temperature, geometric, and sorption effects. Each of these effects imposes certain restrictions on the precision of measurement and on the pressure ranges over which cryopumping may be critically investigated. This report shows how each of these effects may be treated, starting from a definition of the capture coefficient.

SECTION II CRYOPUMPING DEFINITIONS

The pumping speed of a cryosurface can be defined in terms of either a mass flow rate or a volume flow rate (Ref. 1). These are related through the pressure and temperature.

$$\dot{N} = (P_0/RT_0) \dot{V}_0 = \dot{G}/M \quad (1)$$

where

\dot{N} is the flow rate in moles/sec

P_0 is the pressure

T_0 is the temperature

\dot{V}_0 is the volume/sec

R is the universal gas constant in appropriate PVT units

\dot{G} is the mass/sec

M is the molecular weight in the same mass units

In this report, the volumetric pumping speed is used because, in free molecular flow, the theoretical maximum volumetric pumping speed, \dot{V}_{max} , is independent of the pressure. It is given by the product of the theoretical maximum specific pumping speed and the cryosurface area.

$$\dot{V}_{max} = S_{max} A \quad (2)$$

$$S_{max} = \sqrt{RT_g/2\pi M} \quad (3)$$

where

S_{max} is the maximum specific pumping speed in cc/cm² sec

A is the cryosurface area

T_g is the gas temperature

Equation (3) may be derived as follows: from the kinetic theory of gases (Ref. 4) the number of molecules striking a unit area under equilibrium (no flow) conditions is given by

$$\eta = \frac{P}{\sqrt{2\pi mkT_g}} \text{ molecules/cm}^2 \text{ - sec} \quad (4)$$

Where the pressure is in dynes/cm² and the temperature in degrees Kelvin, the Boltzmann constant will be 1.38×10^{-16} ergs/molecule-°K, and the molecular mass will be in gm/molecule.

Dividing both sides by Avogadro's number (6.023×10^{23} molecules/gm mole),

$$\frac{\dot{N}}{A} = \frac{P}{\sqrt{2\pi MRT_g}} \text{ moles/cm}^2 \text{ sec} \quad (5)$$

where

R is in ergs/mole-°K

M is in gm/gm-mole

From the perfect gas law

$$N = PV/RT_g \quad (6)$$

Differentiating with respect to time, at constant temperature and pressure, and dividing by the area gives

$$\frac{\dot{N}}{A} = P\dot{V}_{\max}/RT_g A \quad (7)$$

Combining Eqs. (5) and (7) and replacing \dot{V}_{\max}/A with its equivalent, S_{\max} ,

$$\frac{P}{\sqrt{2\pi MRT_g}} = PS_{\max}/RT_g \quad (8)$$

Rearranging Eq. (8) gives Eq. (3)

$$S_{\max} = \sqrt{RT_g/2\pi M}$$

The criterion for the maximum pumping speed is that every molecule striking the surface will condense. This will not be true in the general case; therefore, it is necessary to define an effective pumping speed.

By the conservation of mass and the continuity condition, it may be stated that (Ref. 1)

$$\frac{P_o \dot{V}_o}{RT_o} = \frac{P\dot{V}}{RT_g} + \frac{\dot{P}V}{RT_g} \quad (9)$$

where the left-hand side represents the rate of flow of gas into a chamber, and the right-hand side is the sum of the moles condensed per second and the rate of change in the number of moles in the chamber volume. In the steady-state condition, \dot{P} is zero so that

$$\frac{P_o \dot{V}_o}{RT_o} = \frac{P\dot{V}}{RT_g} \quad (10)$$

or, rearranging,

$$\dot{V} = \left(\frac{P_o \dot{V}_o}{P} \right) T_g/T_o \quad (11)$$

Equation (11) defines the effective pumping speed.

In the pressure range where the theoretical maximum pumping speed is defined, i. e., in the free molecular flow region, the capture coefficient is defined as the ratio of the measured pumping speed to the theoretical maximum pumping speed; thus,

$$C = \dot{V}/\dot{V}_{\max} \quad (12)$$

$$P = P_1$$

$$\alpha = \left(\frac{Q_1}{Q_2} - 1 \right)$$

SECTION III THE CAPTURE COEFFICIENT

3.1 DEFINITION

In the preceding section, the capture coefficient was defined in terms of the pumping speed of the cryosurface. This definition has meaning only in the free molecular flow regime. In this flow regime, collisions between molecules and collisions between molecules and cryosurface are infrequent compared to molecular collisions with chamber walls. Therefore, the temperature of the gas is effectively that of the chamber walls. In the transition range, the definition given in Eq. (12) breaks down because of the inability to calculate \dot{V}_{\max} .

To circumvent this difficulty, the capture coefficient is defined as the probability of condensation per molecular collision with the surface. By this definition, the capture coefficient becomes completely independent of pressure and is a function only of the compositions and temperatures of the gas and surface. The result is the macroscopic definition of the capture coefficient as the ratio of the measure pumping speed to the calculated pumping speed and the microscopic definition as a condensation probability per molecular collision. These concepts of the capture coefficient, corrected and uncorrected for condensate vapor pressure, are discussed in Section IV.

3.2 TEMPERATURE EFFECTS

In any condensation (or evaporation) of a gas molecule, the process is irreversible. Thus, there is an irreversible energy exchange between a particular molecule and the surface (Ref. 5). A molecule approaching a cold surface will irreversibly lose a certain amount of energy during collision. If the loss is sufficient with respect to the molecule's initial energy, the molecule will condense. Thus, there is a certain minimum average energy of approaching molecules, below which the molecules will condense (Refs. 3, 6, and 7). Molecules having a greater energy will on the average escape. According to the relationships derived by Maxwell and Boltzmann (Ref. 8), the fraction of incident molecules in an equilibrium distribution which has an energy greater than a specified value is given by

$$f = e^{-E/kT_g} \quad (13)$$

where E is the specified minimum energy. The fraction, f , is simply the fraction escaping after a single collision and is thus equal to $1 - C$.

Therefore,

$$1 - C = e^{-E/kT_g} \quad (14)$$

or

$$\log (1 - C) = -E/kT_g \quad (15)$$

Thus, a relationship results which allows the calculation of the capture coefficient at any gas temperature from that at a given temperature (Ref. 2). That is, if the capture coefficient, C , is known for a gas temperature, T_{g1} , at some specific cryosurface temperature, Eq. (15) would be

$$T_{g1} \log (1 - C_1) = -E/k \quad (16)$$

and since E/k is independent of the gas temperature,

$$T_{g2} \log (1 - C_2) = -E/k \quad (17)$$

or

$$1 - C_2 = (1 - C_1) T_{g1}/T_{g2} \quad (18)$$

In this approach, it has been assumed that the minimum energy, E , will not be a function of the gas temperature. However, E will depend on the identity of the gas, the temperature of the surface, and the composition of the surface. This latter dependence gives a theoretical basis for the so-called bare surface effect (Ref. 9) which indicates a change in pumping speed because a metal cryosurface is coated with gas condensate. To eliminate the ambiguity of many possible surfaces, the capture coefficient, unless otherwise specified, usually refers to condensation on a surface covered with condensate of the gas species under test.

There is another assumption in the derivation of Eq. (15) which restricts its application. The assumption of a Maxwell-Boltzmann distribution eliminates the use of Eq. (15) in cases where several different temperatures exist in the gas phase, or when there is a narrow band of molecular velocities being considered such as those from a molecular beam. In these cases, other relations must be worked out to specify the fraction of total incident molecules having an energy greater than the specified minimum.

Some theoretical work is in progress on the effect of surface temperature (Ref. 5). Intuitively, the capture probability would be expected to decrease as the surface temperature increases. This has been found to be true for several gases.

3.3 MIXTURES

Where there is more than one species of gas incident on the cryo-surface, there are two effects which may occur.

1. The capture coefficients of the individual gases may be altered because of the changed composition on the surface.
2. If one or more of the gases is a noncondensable gas, adsorption of the noncondensables and their subsequent covering over with the condensable gases may lead to trapping (Ref. 10). Thus, gas is being pumped by a cryosurface even though its partial pressure in the system is far below its vapor pressure at the surface temperature.

As for the alteration of the capture coefficients because of interaction of different species of condensable gases, present understanding is so limited that each mixture must be studied individually. Various possible types of interaction, from no change to all combinations of increasing and decreasing capture coefficients, have been observed.

SECTION IV TEMPERATURE AND PRESSURE CORRECTIONS

4.1 THERMAL TRANSPIRATION

The thermal transpiration effect is often confusing to the novice in vacuum work. In effect, it is simply a consequence of the conservation of mass and the kinetic theory equations for the number of gas molecules incident on a unit area. The confusion generally arises from application of the thermal transpiration effect to the pressure reading on a hot filament gage where the calibration temperature is different from the real temperature of the gage envelope.

In this section, the general thermal transpiration effect is derived. In Section 4.2.3, it is shown how this effect can be cancelled out by the calibration procedure.

Consider a vessel, V_1 , at a temperature, T_1 , connected by a tube to another vessel, V_2 , at T_2 (Fig. 1). For the sake of simplicity assume that the system, consisting of V_1 , V_2 , and the tube, contains only a single species of gas, is leak free, and without sorption effects. The number of

molecules entering the connecting tube from V_1 will be given by Eq. (4) (Ref. 1),

$$\dot{n}_1 = AP_1 / \sqrt{2\pi mkT_1} \quad (19)$$

and likewise those entering from V_2

$$\dot{n}_2 = AP_2 / \sqrt{2\pi mkT_2} \quad (20)$$

After a sufficient time has elapsed, an equilibrium is reached so that these two equations become equal and

$$\frac{AP_1}{\sqrt{2\pi mkT_1}} = \frac{AP_2}{\sqrt{2\pi mkT_2}} \quad (21)$$

or

$$\frac{P_1}{\sqrt{T_1}} = \frac{P_2}{\sqrt{T_2}} \quad (22)$$

This is the essence of the thermal transpiration effect. Two points are immediately obvious: (1) Eq. (22) only applies in free molecule flow conditions, and (2) the pressures must be constant (Ref. 1).

Consider what happens when V_2 is an external pressure gage at an average temperature of 400°K, and V_1 is a vacuum system at 300°K. The gage is calibrated on the basis of the pressure in V_1 so that the reference temperature for the gage calibration is 300°K with the gage at 400°K. If the gage should be cooled by a draught or heated by inadvertent insulation, the gage calibration will change. Because this effect is proportional to the square root of the gage temperature, it is usually negligible compared to random variations in gage calibration. Increasing the gage temperature to 500°K only increases the pressure reading 10 percent. However, for precision work, a constant gage temperature is desirable.

4.2 VAPOR PRESSURE CORRECTION

To establish the relationship between the capture coefficient, C , and the condensation coefficient, C_g , the effects of the vapor pressure of the gas condensate must be considered under each set of system conditions where measurements are made. In a system in which all the surfaces, chamber walls and cryosurface are at the same temperature, the vapor in equilibrium with the gas condensate will exert a pressure equal to the vapor pressure of the condensate. The number of molecules incident on

a unit area of the cryosurface, per unit time, η , is calculated from kinetic theory and is given by Eq. (23):

$$\eta = \frac{P_s}{\sqrt{2\pi mkT}} \quad (23)$$

A fraction, C_g , of the incident molecules will be condensed on the surface, and a fraction $(1 - C_g)$ will be reflected. However, since this is an isothermal equilibrium process, there can be no net mass or energy transfer (Eq. (21)); therefore, there will be the same number of molecules evaporating as are being condensed. Thus, in this case, the condensation coefficient and the evaporation coefficient will be equal and finite; however, the capture coefficient will be zero. That is, there will be no net reduction in the number of molecules in the gas phase.

4.2.1 Non-Isothermal Zero Flow Case

The system where the chamber wall temperature and the cryosurface temperature differ is presented in Fig. 3. In this case, there is no gas flow into the system, and therefore, after a certain length of time the system will reach equilibrium conditions.

Of the molecules incident on the surface, a certain fraction, C_g , will be condensed and a fraction, $1 - C_g$, will be reflected. Also, a certain number of molecules will be evaporated

$$\frac{C_s P_s}{\sqrt{2\pi mkT_s}} \quad (24)$$

Since at equilibrium, P_g will equal the vapor pressure, P_s , the number of molecules being condensed must equal the number of molecules being evaporated and

$$\frac{C_g P_g}{\sqrt{T_g}} = \frac{C_s P_s}{\sqrt{T_s}} \quad (25)$$

Hence, there will be a net energy transfer but no net mass flow. A comparison of the two sides of Eq. (25) indicates that only under isothermal equilibrium conditions is the evaporation coefficient, C_s , equal to the condensation coefficient, C_g . Also, regardless of the temperature variance in the system, a capture coefficient or net mass transfer will occur only under flow conditions since without flow of molecules into the system, P_g will equal P_s in a relatively short time.

4.2.3 Non-Isothermal, Positive Flow Case

For the system shown in Fig. 3, the effect of the vapor pressure of the condensate on the measurement of the capture coefficient may be determined. It was shown earlier that the capture coefficient by its definition is independent of the pressure so that if the pressure is increased, by flowing gas into the chamber to give a net mass flow, the net number of molecules removed from the system, η_c , by the cryo-pumping action of a unit area of surface will be the difference between the condensation and the evaporation rates.

$$\eta_c = \frac{C_g P_c}{\sqrt{2\pi m k T_g}} - \frac{C_s P_s}{\sqrt{2\pi m k T_s}} \quad (26)$$

where P_c is chamber pressure with flow, and the total number of incident molecules per unit area is

$$\eta_i = \frac{P_c}{\sqrt{2\pi m k T_g}} \quad (27)$$

The ratio, η_c/η_i , will be the capture coefficient, C .

$$\eta_c/\eta_i = C = C_g \left(1 - \frac{C_s P_s}{C_g P_c} \sqrt{\frac{T_g}{T_s}} \right) \quad (28)$$

But from Eq. (25),

$$P_g = \frac{C_s}{C_g} P_s \sqrt{\frac{T_g}{T_s}} \quad (29)$$

Therefore,

$$C = C_g \left(1 - \frac{P_g}{P_c} \right) \quad (30)$$

where P_g is the equilibrium pressure with no gas flow into the chamber. This indicates that when the vapor pressure, P_s , or the equilibrium pressure with no gas flow, P_g , is much less than the steady state chamber pressure, P_c , the capture coefficient is equal to the condensation coefficient.

This relationship between the capture coefficient and the condensation coefficient is also shown experimentally. Recalling the definitions of the experimental pumping speed, \dot{V} , Eq. (11),

$$\dot{V} = \frac{P_o \dot{V}_o T_g}{P_c T_o}$$

and the experimental capture coefficient, C , Eq. (12),

$$C = \frac{\dot{V}}{\dot{V}_{max}}$$

and combining the two equations, the definition for the capture coefficient in terms of experimental parameters is obtained:

$$C = \frac{P_o \dot{V}_o T_g}{P_c \dot{V}_{max} T_o} \quad (31)$$

Equating Eqs. (30) and (31),

$$\frac{P_o \dot{V}_o T_g}{P_c \dot{V}_{max} T_o} = C_g \left(1 - \frac{P_g}{P_c} \right) \quad (32)$$

and solving for C_g ,

$$C_g = \frac{P_o}{P_c - P_g} \frac{\dot{V}_o T_g}{\dot{V}_{max} T_o} \quad (33)$$

An expression for C_g , the condensation coefficient, in terms of experimental parameters is obtained. A comparison of Eqs. (31) and (33) indicates that

$$C = C_g \quad \text{when} \quad P_c \gg P_g \quad (34)$$

A further illumination of the effect of temperature is found based on Eqs. (2) and (3):

$$V_{max} = S_{max} A = A \sqrt{\frac{RT_g}{2\pi M}} \quad (35)$$

Substituting Eq. (35) in Eq. (33),

$$C_g = \frac{P_o \dot{V}_o T_g}{\Delta P A T_o} \sqrt{\frac{2\pi M}{RT_g}} \quad (36)$$

where

$$\Delta P = P_c - P_g \quad (37)$$

Suppose now that the pressure gage was calibrated at T_o so that from Eq. (22) with ΔP_m equal to the measured pressure drop,

$$\Delta P = \Delta P_m \sqrt{\frac{T_g}{T_o}} \quad (38)$$

Substituting Eq. (38) in Eq. (36),

$$C_g = \frac{P_o \dot{V}_o T_g}{\Delta P_m A T_o} \sqrt{\frac{T_o}{T_g}} \sqrt{\frac{2\pi M}{RT_g}} \quad (39)$$

Simplifying, the result is

$$C_g = \frac{P_o \dot{V}_o}{\Delta P_m A} \sqrt{\frac{2\pi M}{RT_o}} \quad (40)$$

Thus, for the same inflow rate, the condensation coefficient is inversely proportional to the measured pressure drop without regard to the gas temperature. This affords a simple means of comparing condensation coefficients at different gas temperatures which is independent of the calibration, so long as the pressure gage is linear and at constant temperature.

SECTION V CHAMBER DESIGN

5.1 GEOMETRIC EFFECTS IN CRYOPUMPING

Another factor to be considered in cryopumping measurements is the geometric effect (Ref. 11). The position of the pressure gage relative to both the cryosurface and the gas inlet as well as the shape of the cryosurface can have important effects on the pumping speed measurement. These effects have been discussed in detail by several investigators (Refs. 12 through 16).

An additional difficulty arises, in some instances, because of the shape of the cryosurface. Errors in the determination of the capture coefficient are encountered when concave cryosurfaces are employed and are caused by successive collisions with the cryosurface without intervening wall collisions. The effect is to increase the pumping speed and the measured value of the capture coefficient. Thus, the apparent capture coefficient of carbon dioxide on a 77°K cryosurface may be increased from 0.6 to 1.0 through the use of parallel fins with a separation less than half their depth (Ref. 11).

At higher pressures, when the mean free path is comparable to the cryosurface dimensions, an interaction between the fins and gaseous impurities becomes evident (Ref. 11). Some of the momentum of the condensing gas is transferred to the noncondensable impurities, causing them to collect between the fins near the cryosurface. This higher concentration of noncondensable impurities interferes with the condensation, and the net result is a reduction in pumping speed. Thus, the pumping speed will be a function of gas purity in the transition and continuum flow regions: the higher the purity, the higher the pumping speed. This effect of impurities becomes negligible at lower pressures and is not detectable in the free molecular flow region. However, at higher pressures, it becomes important even for convex cryosurfaces without fins (Ref. 17).

5.2 REFRIGERANT LINES

An important experimental problem in the measurement of cryopumping rates is the possibility of the occurrence of condensation on surfaces, adjacent to the defined cryosurface, which have been cooled by conduction and radiation. This condensation will obviously lead to errors in the measurement of the specific cryopumping speed because of the change in effective cryosurface area with pressure. This problem is particularly troublesome when the cryosurface is precoated with the condensable gas in order to eliminate the effects of surface contamination and bare surface effects (Ref. 17). The precoating is usually accomplished at relatively high pressures, so that the warmer surfaces may become rather heavily coated with cryodeposit. This leads to an increase in the background pressure, since the vapor pressure of the cryodeposit will be determined by the temperature of the surface. For example, it can be shown that the pressure of an 87°K surface amounting to only one percent of the area of the 77°K surface can more than double the background pressure in the chamber. The error that would be introduced into the capture coefficient would depend upon the ratio of the pressures with and without flow (Eq. (30)).

SECTION VI INSTRUMENTATION

A system parameter, as considered in this report, is any factor or quantity which may influence the values of the experimental quantities to be determined, namely the pumping speed or capture coefficient. The system parameters of major importance are pressure, temperature, sorption, system outgassing and conductance. A discussion of each parameter and some of the interactions follows.

6.1 PRESSURE

The pressure is the most important experimental parameter that must be measured. As shown in Eq. (2), two different pressure measurements are required: (1) the pressure behind the standard leak, P_0 , and (2) the steady-state chamber pressure, P_c . Normally, P_0 is 760 mm Hg and may be measured with a mercury manometer. The accuracy of this measurement is about 0.1 percent or better. Also, P_0 is not influenced by chamber conditions; however, P_c is dependent on essentially all of the system parameters. To show this dependency, an actual cryopumping experiment must be considered.

The pressure-time curve for a pure condensable gas with negligible chamber outgassing and leakage is indicated in Fig. 4. Starting at some initial chamber pressure, A, the test gas is introduced into the system at a constant known flow rate. The pressure in the system increases as a function of time as indicated by line AB in Fig. 6. At point B, the pumping speed of the cryosurface equals the rate of introduction of the condensable test gas, and a steady state is reached as shown by line BC. If at point C the inflow is stopped, the pressure will drop to D, which is the initial pressure, A. The drop is caused by the cryosurface removing the condensable gas from the system and is a direct measure of ΔP_m in Eq. (40). With a pure gas (no noncondensables), the experiment is quite straight-forward, and the steady-state pressure measurements may be made with any properly calibrated instrument.

If the test gas contains any noncondensables as impurities, the chamber pressure will follow a different path, shown as the dotted line in Fig. 4. From the initial pressure, A, the chamber pressure increases along AB as the test gas is introduced. At B, a steady state is established between the condensable gas and the pumping speed of the cryosurface; however, the chamber pressure continues to rise along BF because of the introduction of noncondensable impurities. When the inflow is stopped at F, the pressure will fall to a new steady-state value, H. If the noncondensables are caused entirely by the inflow through the standard leak, the chamber pressure will remain constant along line HG. If, however, part of the noncondensables were caused by outgassing from the chamber walls or leakage, the pressure will increase along HI. The pressure drop FH is an actual measure of the steady-state pressure of the condensable gas. This can be shown by considering a system with a 10°K cryosurface and a test gas, nitrogen (N₂), that contains 5 percent helium (He) as a noncondensable impurity. The pressure at F (Fig. 4) would be

$$P_F = P_{N_2} + P_{He} \quad (41)$$

where P_F is the total pressure in the system, and P_{N_2} and P_{He} are the partial pressures of nitrogen and helium, respectively. Substituting for the partial pressures,

$$P_F = \alpha_{N_2} (0.95) r_F + \alpha_{He} (0.05) r_F \quad (42)$$

where r is the pressure gage reading, the decimals are the fractions of the gases present, and α_{N_2} and α_{He} are the gage sensitivities for the gases indicated.

The pressure at H, however, is caused only by the noncondensables since the capture coefficient of N₂ on a 10°K cryosurface is 1.0.

Then

$$P_H = \alpha_{He} r_H \quad (43)$$

If all the noncondensables introduced remain in the system, then

$$\alpha_{He} r_H = \alpha_{He} (0.05) r_F \quad (44)$$

and

$$P_F - P_H = \alpha_{N_2} (0.95) r_F \quad (45)$$

Even if the capture coefficient were not unity, the pressure drop, $P_F - P_H$, would still be a valid measure of the steady-state pressure of the condensable gas under the conditions stated.

If more than one impurity were present, Eq. (41) would simply be

$$P_F = P_{N_2} + \sum_i^n P_i \quad (46)$$

where P_i is the partial pressure of the "i"th component. However, if the noncondensables were absorbed or trapped by the condensate, Eqs. (42) and (44) would not be valid, and an error would be introduced if Eq. (45) were used in calculating the pumping speed or capture coefficient for the system. The magnitude of this error would depend on the rates of absorption or trapping of the noncondensables by the cryodeposit. These rates are dependent on the particular noncondensable gases present, the condensate, and the system conditions, such as total pressure, partial pressures of each constituent, and cryosurface temperature. These rates are also a function of the rate at which new condensate surfaces are formed. Some of the possible errors in the pressure measurements can be minimized by employing a mass spectrometer as the pressure measuring device. This is because the partial pressure of the condensable gas may be followed, and the measured drop would not generally be influenced by the noncondensables present.

6.2 CALIBRATION OF ION GAGES AND MASS SPECTROMETER

The most common method of calibrating an ion gage is the pressure rise method. This method consists of introducing a known gas into a chamber of volume V_c through a standard leak, \dot{V}_o , which has been calibrated at a given temperature and pressure, T_o and P_o . The pressure rise in the chamber, \dot{P}_c , can be calculated from

$$\frac{P_o \dot{V}_o}{T_o} = \frac{\dot{P}_c V_c}{T_c} \quad (47)$$

where T_c is the chamber temperature. The measured rate of pressure rise from the ion gage, \dot{P}_m , is then corrected to the true rate of rise,

\dot{P}_c , by employing a correction or sensitivity factor, α .

$$\dot{P}_c = \alpha \dot{P}_m \quad (48)$$

The factor, α , will be different for each gas and normally will be different for different ion gages. There are a number of factors that affect the sensitivity of an ion gage. (A thorough discussion of these factors is given in Ref. 18). The gage readings and the method itself are subject to several sources of error, some of which have already been discussed in previous sections of this report.

The pressure rise method is also used in calibrating mass spectrometers. However, the sources of error caused by outgassing and sorption may be greatly reduced by saturating the system with a gas other than that to be used in the calibration. This method is effective because the mass spectrometer may follow any given mass (or partial pressure) rather than total chamber pressure, and the desorption is dependent on the immediate past history of the chamber.

Another method of calibrating ion gages and mass spectrometers is the two-volume expansion method. In a system such as shown in Fig. 1 where the two volumes are separated by a large vacuum valve, volume one, V_1 , is 0.1 liter and volume two, V_2 , is 100 liters. Then, if the pressure in V_1 were regulated and accurately measured with a McCleod gage from 1 micron to 1 mm, the pressure in V_2 could be varied from 10^{-6} to 10^{-3} and used to calibrate the ion gage or mass spectrometer.

6.3 TEMPERATURE MEASUREMENT AND CONTROL

The temperatures that must be considered in cryopumping experiments are the standard leak temperature, T_O , chamber wall temperature, T_C , gas temperature, T_g , cryosurface temperature, T_S , and the pressure gage temperature, T_a . Since free molecular flow conditions exist in the chamber and the ratio of chamber wall to cryosurface area is large for most systems, T_C and T_g are the same. Also, the gage calibration corrects the gage temperature to room temperature, making T_O and T_a equal. Therefore, the only temperatures that actually must be measured are T_O , T_g , and T_S .

Room temperature, T_O , can be measured very accurately, and a discussion of this measurement is not necessary; however, as pointed out previously, the measurement should not be neglected in precise work.

The measurement of the gas temperature, T_g , consists mainly of an accurate measurement of the chamber wall temperature. This is normally

accomplished with several thermocouples spaced at appropriate intervals along the length and around the circumference of the vacuum chamber in order to measure temperature uniformity. The thermocouples are, of course, calibrated for the appropriate temperature range.

One of the better methods of obtaining uniform wall temperature is to use a coolant which has a phase change at the desired temperature, for example, liquid nitrogen at 77°K, solid carbon dioxide at 195°K, water at 0°K, etc. In the past (Refs. 2 and 6), the 77 and 195°K wall temperatures were the main temperatures studied below room temperature; however, current vacuum chamber designs incorporate shrouds for use in conjunction with refrigeration systems to obtain any desired temperature down to and even below 77°K. In these systems, thermocouples in the inflow and outflow lines or on the shroud itself would be required for gas temperature estimation. Since the gas temperature enters the calculations only as a square root, only a small error would be introduced if the actual measurement were off a few degrees. The cryosurface temperature measurement is much more critical than the gas temperature. A few degrees error in its measurement could amount to as much as 50 percent of the temperature. As with the wall temperature, the cryosurface temperature may be controlled and measured by using a coolant which has a phase change at the desired temperature such as the boiling points of He - 4.2°K, H₂ - 20.4°K, and N₂ - 77°K. For intermediate temperatures, a helium refrigeration system may be employed. Temperatures from 10 to 30°K are obtained with this type of system without the use of complicated transfer systems. These temperatures are usually measured with a helium gas or a hydrogen vapor pressure thermometer. For temperatures between 30 and 77°K, the same type of refrigeration system may be used, but a controlled heat load must be introduced into the system. This type of cryosurface temperature controller is now in use at Arnold Center.

When it is required that cryosurface temperatures in the range from 10 to 30°K be measured, most thermocouples become unreliable, and thermistors (gold-cobalt thermocouples) or resistance thermometers may be used. If the system must be bakeable to 400°C, either a gas or a vapor pressure thermometer must be used. (Information concerning a new gold-iron thermocouple with a range from 4 to 500°K is found in Ref. 19). Because of the large bulb size required for gas thermometers, the small bulb vapor pressure thermometer, using hydrogen from 12 to 30°K, has come into use. The principle of the vapor pressure thermometer is that the gas will condense in the sensing bulb, and the pressure read by a gage connected to this bulb will equal the vapor pressure of the gas at the temperature of the condensed phase. Although the idea is simple, the design of the thermometer is not. There is no problem with thermal transpiration if the pressure is higher than 1 torr since the finest tubing generally used

has an inside diameter greater than 1 mm which is many times greater than the mean free path of the hydrogen. If finer tubing is used, the range of the thermometer may be restricted. Although the tubing diameter has little effect of itself, it can be shown that the volume of the external system is important. If the volume is too large, the bulb will fill with condensed hydrogen, and the interface between the condensed and gas phases will be located in the tube at a different temperature from the bulb so that the pressure will not accurately reflect the bulb temperature.

Most thermometers necessarily measure a temperature inside the cryosurface. The capture coefficient, however, will depend on the effective temperature of the cryodeposit. Consequently, for reliable capture coefficient measurements, it must be shown that the temperature difference is negligible between the cryodeposit surface and a hydrogen vapor pressure thermometer within the cryosurface.

SECTION VII

SUMMARY

In this report, some of the factors affecting the measurement of pumping speeds and capture coefficients have been discussed. Methods of data treatment have been presented which are of assistance in the elimination of spurious results. The necessity for attention to detail in order to obtain meaningful results cannot be overemphasized.

The information presented in this report result from the cumulative evidence of three years devoted to the study of all aspects of cryopumping at Arnold Center. Work on the three main problems of cryopumping (directed flow cryopumping, the correlation of capture coefficients with physical properties, and the effect of cryosurface temperature on the capture coefficient) is continuing. The theoretical basis of these problems lies in the exact nature of the irreversible energy exchange between a gas molecule and a surface.

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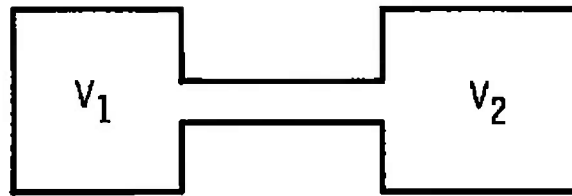


Fig. 1 Two-Volume System for Thermal Transpiration

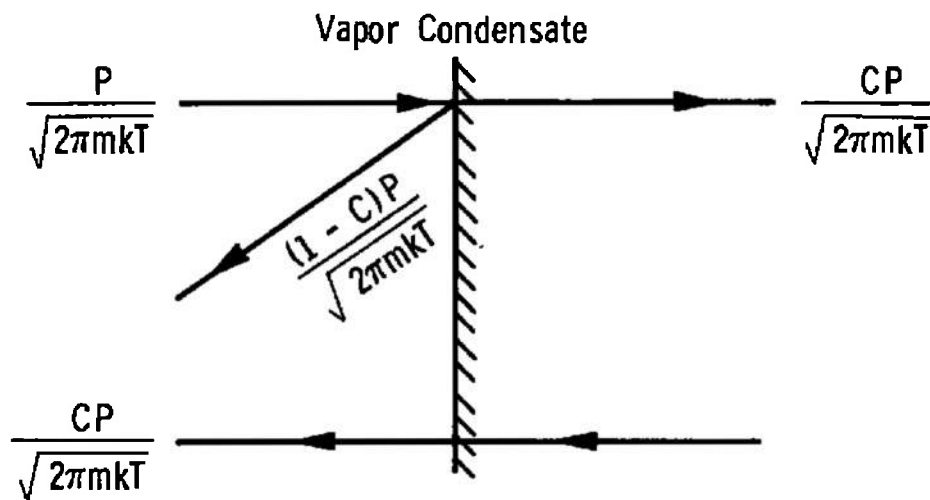


Fig. 2 Isothermal Capture Coefficient

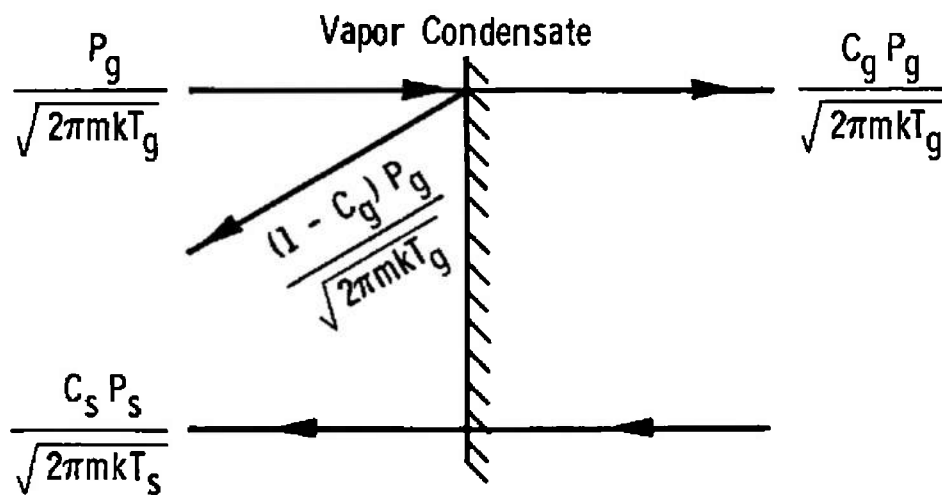


Fig. 3 Non-Isothermal Capture Coefficient

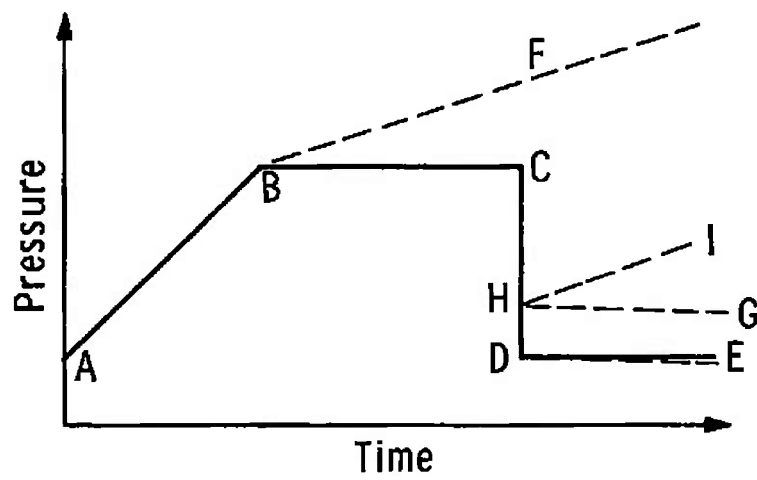


Fig. 4 Schematic of the Pressure-Time Curve

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